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Indian Standard

**METHODS FOR MEASUREMENT OF
AIR POLLUTION**

**PART XVIII CONTINUOUS ANALYSIS
AND AUTOMATIC RECORDING OF THE OXIDANT
CONTENT OF THE ATMOSPHERE**

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**BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002**

*Indian Standard***METHODS FOR MEASUREMENT OF
AIR POLLUTION****PART XVIII CONTINUOUS ANALYSIS
AND AUTOMATIC RECORDING OF THE OXIDANT
CONTENT OF THE ATMOSPHERE**

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*Indian Standard***METHODS FOR MEASUREMENT OF
AIR POLLUTION****PART XVIII CONTINUOUS ANALYSIS
AND AUTOMATIC RECORDING OF THE OXIDANT
CONTENT OF THE ATMOSPHERE****0. FOREWORD**

0.1 This Indian Standard (Part XVIII) was adopted by the Indian Standards Institution on 26 September 1974, after the draft finalized by the Air Pollution Sectional Committee had been approved by the Chemical Division Council.

0.2 This method is based on ASTM D 2011-65 'Method of test for continuous analysis and automatic recording of the oxidant content of the atmosphere', issued by the American Society for Testing and Materials, Philadelphia (USA).

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part XVIII) covers the determination of low concentrations of oxidants in the atmosphere. The term 'oxidants' as used in this standard refers to substances capable of liberating iodine from buffered, neutral potassium iodide solutions. The method is quantitative for ozone. Nitrogen dioxide and certain peroxidized compounds are also registered, but with low sensitivity. Reducing substances such as sulphur dioxide or hydrogen sulphide interfere with the determination by consuming iodine.

1.2 The recorders are generally set up to indicate the range of 0 to 20 to 100 ppm of oxidant (ozone) on a logarithmic scale. This range can be modified by changing the gas-to-liquid ratio in the scrubber or the dimensions of the optical cell, or both.

*Rules for rounding off numerical values (revised).

2. TERMINOLOGY

2.1 For the purpose of this standard, definitions given in IS : 4167-1966* shall apply.

3. OUTLINE OF THE METHOD

3.1 Air samples containing oxidant are scrubbed countercurrently in a wetted wall absorber by a 2 percent solution of buffered, neutral potassium iodide in water. The yellow colour of the resulting solution is measured photometrically and recorded continuously on a strip-chart recorder. As long as a constant air to liquid ratio is maintained, the instrument can be calibrated to read directly the concentration of a reference oxidant, such as ozone. Known mixtures of ozone and air or known concentrations of iodine in potassium iodide solution may be employed for this calibration. The absorbing solution is regenerated by passing it through a bed of activated carbon immediately before reuse.

4. INTERFERENCES

4.1 Any substance capable of reacting rapidly with a neutral potassium iodide solution containing free iodine, will register on the recorder, either as an increase or a decrease of the reading. In practice, only ozone, nitrogen dioxide, some peroxidized organic compounds and chlorine liberate iodine, while sulphur dioxide, or hydrogen sulphide reduce it and thus lower the reading. If reducing substances are present, they shall be measured independently and appropriate corrections applied.

5. APPARATUS

5.1 The apparatus shall consist essentially of two functional parts, namely, a continuous gas-liquid contacting device, and a recording colorimeter as illustrated in Fig. 1.

5.1.1 Liquid System — The liquid system shall consist of a 4-litre bottle *A* from which the reagent solution is forced by a constant-delivery pump *B*, *C*, *D*, through a bed of 2.36 mm to 850 micron activated carbon *E* to the top of the scrubbing column *H*. It is optional whether or not the solution passes through a reference colorimeter cell *F*₁ attached to the light source *G*. The solution shall then flow down over the inside wall of the glass tube *H* having a 6-mm inside diameter and 500 to 600 mm long. A 6-pitch spiral of 1-mm glass rod shall fit snugly against this wall increasing the area of the absorbing surface, and assuring that this surface is completely and continuously wetted.

5.1.1.1 An alternative method of dispensing the absorbent to glass tube *H* is illustrated in the insert in Fig. 1. An excess of liquid shall be delivered to the constant-level reservoir *R*, the excess returning to the supply bottle through the overflow *S*. Constant delivery to glass tube *H* shall be accomplished by means of the stopcock *K* and the rotameter *T*₂.

*Glossary of terms relating to air pollution.

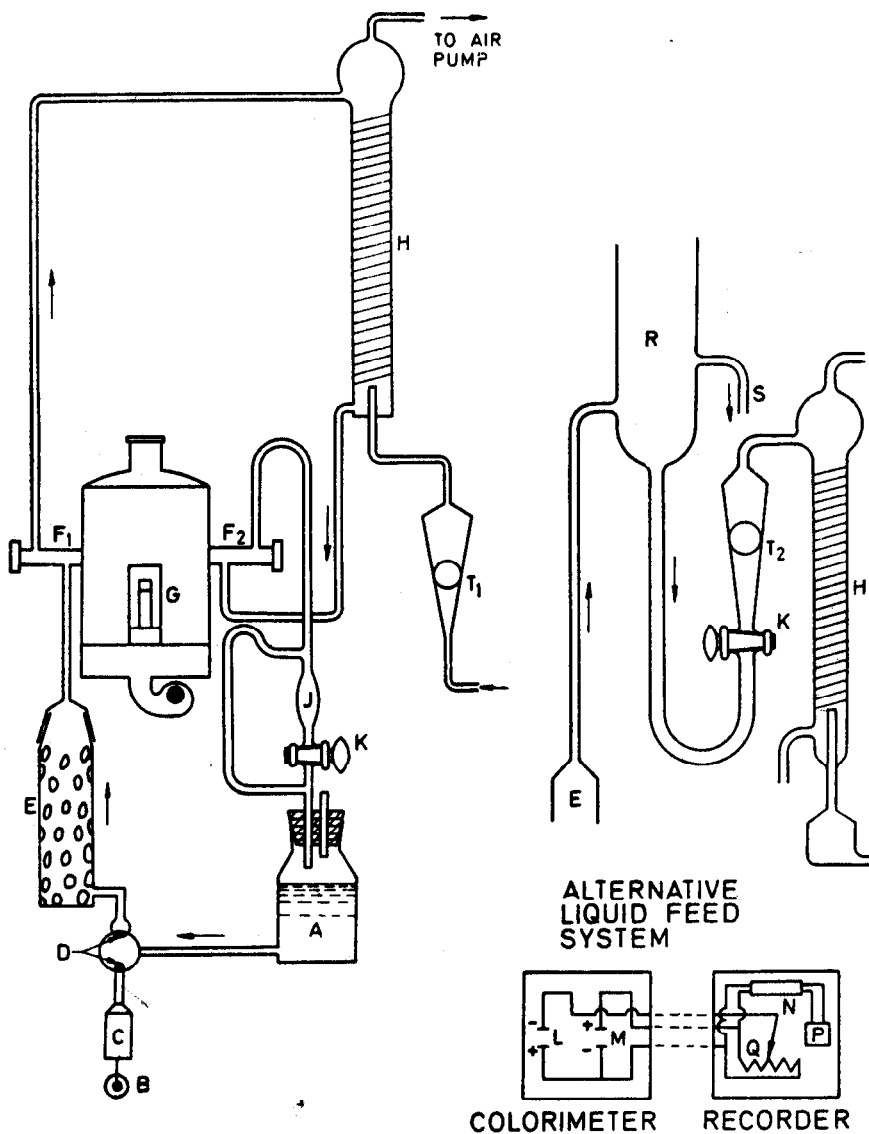


FIG. 1 OXIDANT RECORDER SHOWING LIQUID AND GASEOUS FLOW SYSTEMS, PHOTOMETER, AND WIRING DIAGRAM OF COLORIMETER AND RECORDER

5.1.2 The air sample shall be drawn through the rotameter T_1 into the bottom of the absorber column H by a pump connected to the top of the column. A surge jar prevents accidental entry of the scrubbing solution into the air pump. The solution is thus contacted countercurrently by the air sample. The air flow shall be regulated by an adjusting valve on a by-pass to the pump and also by a needle valve in the main air stream. Alternatively, a reliable gear pump may be employed that gives excellent control of the air stream when properly oiled. From the absorber the solution flows by gravity through the measuring cell F_2 of the recording colorimeter. It then returns to the storage bottle through pipette J and stopcock K which permit periodic measurement of the liquid flow rate when stopcock K is temporarily closed. The time required to fill pipette J shall be noted.

NOTE — Glass should be used for all sampling lines, both gas and liquid. Spherical glass joints or TFE-fluorocarbon connectors are preferred, but vinyl sleeves on butt joints are permissible.

5.1.3 Recording Colorimeter — The recording colorimeter shall be operated at 3600 Å with a bandpass of 400 Å, obtained by passing the light from a medium pressure mercury vapour lamp, through one or two ultraviolet filters. Alternatively, an incandescent lamp can be used. When operated at 7.5 V this lamp has a life of 10 000 hours, but has only 700 hours at 8.5 V, if more ultraviolet light is required to operate the recorder. Figure 1 shows the wiring diagram of a colorimeter using selenium photocells. The lamps, photocells, and recorder are matched so that there is less than 0.5 percent dead band on the recorder. The drift of the colorimeter-recorder system is less than 2 percent in 24 hours with the optical cells dry or removed. There should be provision for adjusting the zero (matched light beam) signal level by ± 30 percent. There may also be provision for adjusting the infinity end of the scale to enable a close match to the calibration data. Optical cell length between 1 and 3 cm is permissible. The cells are designed so that bubbles cannot be trapped but quickly pass through the cells.

5.1.3.1 The recorder indicates absorbance of the solution. This reading can be converted into concentration of iodine in the solution or directly into concentration of ozone in the atmosphere, since the air and liquid flow rates are known and constant and a stoichiometric proportion exists between ozone and iodine.

6. ABSORBENT

6.1 The absorbent shall consist of a potassium iodide (KI) solution (2 percent) buffered to a pH of 7 ± 0.2 by adding disodium phosphate dodecahydrate (3.6 percent) and potassium dihydrogen phosphate (1.4 percent). The pH level shall be maintained at 7 ± 0.2 by adjustment with sodium hydroxide (1 percent) or phosphoric acid (1 percent) solutions. The concentration of the solution increases gradually due to

evaporation of water; this is corrected at weekly intervals, or as necessary, by the addition of distilled water. A wide variation of the potassium iodide concentration can be tolerated.

6.2 The absorbent shall be regenerated during each pass through the system by contacting it with a bed of granular activated carbon that removes all iodine produced by the previous pass. The activated carbon will eventually be exhausted and has to be changed when the filtered solution gets appreciably yellow. A slight yellow tinge of the solution will normally be present in the storage bottle.

6.3 Zeroing is best accomplished by shutting off the air stream and operating the instrument with unaerated solutions. A timer can be used to shut off the air stream for about 1 hour (for example, between 0 400 to 0 500 h) to allow the instrument to equilibrate itself and indicate a zero level.

7. CALIBRATION

7.1 Dynamic calibration of the instrument may be performed by preparing a large volume of a mixture of ozone in air. For this purpose, the output of an ozonizer up to 1 to 2 ml of ozone per minute shall be fed into the intake of a small blower delivering 1 000 to 2 000 litres of air per minute. The gas mixture emerges through a short length of pipe. One portion of this mixture shall be analyzed in the recorder and another aspirated through a faintly blue iodine solution containing starch (0.2 percent) and potassium iodide (0.1 percent). This solution shall be titrated with sodium thiosulphate (0.01 N or 0.001 N) after passage of a measured volume of sample. The end point shall be established by comparison with the unaspirated faintly blue starch-iodine solution.

7.2 Mixtures of ozone and air may be prepared in polyester film bags and admitted to the analyzer through the normal sampling circuit. The bags should have a capacity of about 100 litres. They shall be tested for leaks and perfectly dry and clean inside. Ozone mixtures can be prepared conveniently by passing air through a tube enclosing a small mercury lamp. The concentration of ozone depends on the flow rate, the size of ballast lamp used in series with the mercury bulb, and the voltage applied. The mixtures shall be analyzed at the time they are used since the ozone decomposes gradually. The analysis can be performed by passing a measured volume through an impinger containing buffered potassium iodide (KI) solution and determining the released iodine by titration or by a spectrometer method. With recorders that have an infinity adjustment, make the first mixture about ten times the midscale concentration and adjust the upper end of the recorder scale, having previously adjusted to zero with pure air. Make the second mixture about midscale concentration and adjust sample or reagent flow rates, or both, until the recorder reading agrees with the absorbance scale template according to the analytically determined concentration of the ozone. Check several other points on the curve if desired.

7.3 The colorimeter can also be calibrated by passing the absorbent to which measured concentrations of iodine have been added, through the optical cell.

7.4 The calibration chart of the instrument can be constructed from the data given in 7.1, 7.2, or 7.3. Either a linear or logarithmic chart may be employed. A scale or calibration curve can be constructed to convert either chart to read in pphm. For recorders equipped with infinity adjustment, the logarithmic chart can be made to read directly in pphm by adjusting the flow rates of gas or liquid, or both, within the operation limits of the absorber.

8. PROCEDURE

8.1 Place a fresh absorbent solution in the storage bottle and operate the solution pump until the liquid lines are full. After the flow has stabilized itself, zero the recorder and start the air pump. Set the flows at convenient rates such as 4 ml/min of solution and 4 l/min of air.

8.2 Check and adjust the flow rates daily. Adjust the pH of the solution once or twice a week, at which time the solution shall be brought up to volume by addition of distilled water.

8.3 Change the carbon filter about once a month, depending on its size and the quality of the carbon. Change the absorbing solution at the same time.

9. PRECISION

9.1 Experience has shown that different instruments of this design will agree with each other within ± 5 percent of the average concentration at midscale.

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